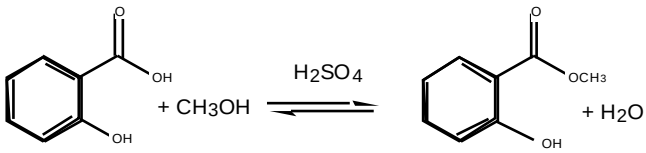
O-12. Matching the reason with the instruction: Making methyl salicylate

Match the reason on the right with the instruction on the left:



Dissolve 4 g of salicylic acid in 25 mL of methanol in a **dry** 50 mL round bottomed flask.

Carefully **add 5 mL of concentrated sulfuric acid**, with gentle swirling.

Add one or two boiling chips and equip the flask with a reflux condenser.

Reflux the mixture using a water bath for 1.5 hours.

Cool the flask...

...and then pour the mixture into a large separating funnel containing about 50 mL of cold water and **10 mL of dichloromethane**, shake, and run out (and retain) the organic extract after the layers have separated.

Repeat the extraction with a fresh 10 mL portion of dichloromethane, combine the two organic extracts and discard the aqueous layer.

Place the combined dichloromethane extracts back into the separating funnel and **wash with about 10 mL of 10% sodium bicarbonate solution.**

Again, discard the aqueous layer. **Dry the organic extract over a little anhydrous Na_2SO_4**, filter, and evaporate the filtrate (add a boiling chip) on the steam bath to leave an oil.

Filter the ester through a small filter funnel **containing a plug of cotton wool** into a clean, dry, tared sample tube. Record the yield of methyl salicylate.

Concentrated sulfuric acid acts as a catalyst in the esterification. Also, it has powerful dehydrating properties so will remove the water produced and draw the reaction to the right and so increase the amount of product.

The mixture is refluxed to speed up the reaction, while preventing the loss of solvent and a water bath is used as it helps minimise the risk of the methanol igniting.

The ester is filtered to remove any pieces of boiling chip, so a plug of cotton wool is sufficient.

The dichloromethane will contain some water from the extraction process, and this is best removed at this stage, as it can't be boiled off on the water bath (obviously). The anhydrous Na_2SO_4 absorbs water to form $Na_2SO_4,10H_2O$.

As you can see from the equation above, water is one of the products of the reaction. The equilibrium constants for esterifications of most primary alcohols are around unity, so the presence of some water in your reaction mixture will significantly reduce your yield. Methanol acts as both the solvent and a reagent. Its presence in large excess helps drive the reaction forward.

The ester is more soluble in dichloromethane than it is in water. By dissolving in dichloromethane, the ester is separated from other components of the reaction mixture which are more water soluble.

Boiling chips help the mixture to boil evenly, preventing bumping (uneven, "explosive" boiling).

Despite the fact that the ester is more soluble in dichloromethane than the acid, the dichloromethane will still contain appreciable quantities of the acid that must be separated from the ester. Washing with sodium bicarbonate will convert the acid into its sodium salt, which is NOT soluble in dichloromethane, achieving the separation.

The flask should be cooled before it is poured into the separating funnel as the reaction mixture is highly acidic, and would produce a significant amount of heat when added to the contents of the flask. If the reaction mixture is hot, the addition may be dangerous.

The extraction is repeated to ensure as little product as possible remains in the aqueous layer, which can then be discarded.

ANSWERS

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